SOP NO. HW-36/Pesticide Data Validation
USEPA Contract Laboratory Program
Statement of Work for Organic Analysis of Low/Medium Concentration of Pesticide Organic Compounds SOM01.1

Prepared by: George Karras Date: 12/05/06
George Karras, Chemist
Hazardous Waste Support Section

Peer Reviewed by: Russell Arnone Date: 12/05/06
Russell Arnone, Chemist
Hazardous Waste Support Section

Concurred by: Linda Mauel Date: 12/5/06
Linda Mauel, Chief
Hazardous Waste Support Section

Approved by: Robert A. Runyon Date: 12/11/06
Robert Runyon, Chief
Hazardous Waste Support Branch

Annual Review

Reviewed by: _____ Date: _____
Name

Reviewed by: _____ Date: _____
Name

TABLE OF CONTENTS

INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to the method in the "USEPA Contract Laboratory Program Statement of Work for Organics Analysis Multi-Media, Multi-Concentration, SOM01.1, May 2005". The validation procedures and actions discussed in this document are based on the requirements set forth in the "USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, January 2005". This document attempts to cover technical problems specific to low/Medium concentration of Pesticide compounds. Situations may arise where data limitations must be assessed based on the reviewer's own professional judgement.

In addition to technical requirements, contractual requirements may also be covered in this document. While it is important that instances of contract non-compliance be addressed in the Data Assessment, the technical criteria are always used to qualify the analytical data.

Summary

To ensure a thorough evaluation of each result in a data case, the reviewer must complete the checklist within this SOP, answering specific questions while performing the prescribed "ACTIONS" in each section. Qualifiers (or flags) are applied to questionable or unusable results as instructed. The data qualifiers discussed in this document are as follows:

Data Qualifiers

U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

N - The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."

JN - The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.

UJ - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

R - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Lab Qualifiers:

D - The positive value is the result of an analysis at a secondary dilution factor.

B - The analyte is present in the associated method blank as well as in the sample. This qualifier has a different meaning when validating inorganic data.

E - The concentration of this analyte exceeds the calibration range of the instrument.

P - Pesticide/Aroclor target analytes when the % Difference between the analyte concentrations obtained from the two dissimilar GC columns is greater than 25%.

The reviewer must prepare a detailed data assessment to be submitted along with the completed SOP checklist. The Data Assessment must list all data qualifications, reasons for qualifications, instances of missing data and contract non-compliance.

Reviewer Qualifications:

Data reviewers must possess a working knowledge of the USEPA Statement of Work SOM01.1 and National Functional Guidelines mentioned above.

YES NO N/A

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:______________________ **LAB**:_________________________________

SITE NAME:________________________ **SDG No(s).**:__________________________

1.0 Chain of Custody and Sampling Trip Reports

1.1 Are the Traffic Reports/Chain-of-Custody Records present for all samples? [ ] ___ ___

ACTION: If no, contact RSCC, or the TOPO to obtain replacement of missing or illegible copies from the lab.

1.2 Is the Sampling Trip Report present for all samples? [ ] ___ ___

ACTION: If no, contact either RSCC or ask the TOPO to obtain the necessary information from the prime contractor.

2.0 Data Completeness and Deliverables

2.1 Have any missing deliverables been received and added to the data package? ___ [ ] ___

ACTION: Contact the TOPO to obtain an explanation or resubmittal of any missing deliverables from the lab. If lab cannot provide them, note the effect on the review of the data package in the Contract Problems/Non-compliance section of the Data Assessment.

2.2 Was SMO/CLASS CCS checklist included with the package? [ ] ___ ___

		YES	NO	N/A
2.3	Are there any discrepancies between the Traffic Reports/Chain-of-Custody Records, and Sampling Trip Report?	___	[ ]	___

ACTION: If yes, contact the TOPO to obtain an explanation or resubmittal of any missing deliverables from the laboratory.

3.0 <u>Cover Letter SDG Narrative</u>

		YES	NO	N/A
3.1	Is the SDG Narrative or Cover Letter Present?	[ ]	___	___
3.2	Are case number, SDG number and contract number contained in the SDG Narrative or cover letter (see SOW, Exhibit B, section 2.5.1)? EPA sample numbers in the SDG, detailed documentation of any quality control, sample, shipment, and/or analytical problems encountered in processing the samples? Corrective action taken?	[ ]	___	___
3.3	Does the Narrative contain the following information SOM01.1, page B-12, section 2.5.1)? column used, storage of samples, case#, SDG#, analytical problems, and discrepancies between field and lab weights.	[ ]	___	___
3.5	Did the contractor record the temperature of the cooler on the Form DC-1, Item 9 - Cooler Temperature, and in the SDG Narrative?	[ ]	___	___
3.6	Does the Case Narrative contain the "verbatim" statement (page B-12, section 2.5.1 of the SOM)?	[ ]	___	___

ACTION: If "No", to any question in this section, contact the TOPO to obtain necessary resubmittals. If unavailable, document under the Contract Problems/ Non-Compliance section of the Data Assessment.

	YES	NO	N/A

4.0 Data Validation Checklist

4.1 Check the package for the following (see SOM reporting requirements, section 2.1, page B-10):

	YES	NO	N/A
a. Is the package paginated in ascending order starting from the SDG narrative?	[ ]	___	___
b. Are all forms and copies legible?	[ ]	___	___
c. Assembled in the order set forth in the SOW?	[ ]	___	___
d. All Pesticide Data present?	[ ]	___	

PART A: Low/Medium Pesticide Analyses

1.0 Sample Conditions/Problems

	YES	NO	N/A
1.1 Do the Traffic Reports/Chain-of-Custody Records, Sampling Trip Report or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?	___	[ ]	___

ACTION: If samples were not iced or the ice was melted upon arrival at the laboratory and the temperature of the cooler was > 10^{o} C, then flag all positive results with a "J" and all non-detects "UJ".

2.0 Holding Times

	YES	NO	N/A
2.1 Have any Pesticide technical holding times, determined from date of collection to date of analysis, been exceeded?	___	[ ]	___

2.2 Preservation: Aqueous and Non-aqueous samples must be cooled at 4°C ± 2°C.

ACTION: Qualify sample results according to the following table.

Holding Time Actions for Low/Medium Pesticide Analyses

Matrix	Preserved	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated Compounds
Aqueous	No	≤ 7 days (extraction) < 40 days (analysis)	J*	UJ*
	No	> 7 days (extraction) > 40 days (analysis)	J	UJ
	Yes	≤ 7 days (extraction) ≤ 40 days (analysis)	No qualification	
	Yes	> 7 days (extraction) > 40 days (analysis)	J	UJ
	Yes/No	> 28 Days (Gross Exceedance)	J	R
Non-aqueous	No	≤ 14 days (extraction) ≤ 40 days (analysis)	J*	UJ*
	No	> 14 days (extraction) > 40 days (analysis)	J	UJ
	Yes	≤ 14 days (extraction) ≤ 40 days (analysis)	No qualification	
	Yes	> 14 days (extraction) > 40 days (analysis)	J	UJ
	Yes/No	> 28 Days (Gross Exceedance)	J	R

* Only if cooler temperature exceeds 10°C (see ACTION in Section 1.1 above).
No action required if temperature ≤ 10°C.

3.0 <u>Surrogate Recovery (Form II Pest-1, Form II Pest-2, Form VIII)</u>

3.1 Are the Pesticide Recovery Summary Forms present? [ ] ___ ___

ACTION: Contact the TOPO to obtain an explanation/resubmittal from the lab. If missing deliverables are unavailable, document the effect in the Data Assessment.

3.2 Were the two surrogates, tetrachloro-m-xylene (TCX) and decachlorobiphenyl (DCB) added to all samples, MS/MSD, LCS, blanks including standards? [ ] ___ ___

ACTION: If no, use professional judgment in qualifying data as missing surrogate analyte may not directly apply to target analytes.

3.3 Were outliers marked with an asterisk on Form II? [ ] ___ ___

ACTION: Circle all outliers with a red pencil.

If yes, were effected samples re-analyzed? [ ] ___ ___

3.4 The RTs of the surrogates in each Performance Evaluation Mixture (PEM), mid-point Individual Standard Mixture (A and B) or (C) used for continuing calibration verification, all samples, including MS/MSD, LCS and all blanks must be within the calculated RT window. TCX must be within ± 0.05 minutes and DCB must be within ± 0.10 minutes of the mean retention time (RT) determined from the initial calibration and tabulated in Form VIII Pest.

Were any outliers marked with an asterisk on Form VIII Pest? ___ [ ] ___

ACTION: Circle all outliers with a red pencil. If any Surrogate is outside the required limits, qualify their associated target compounds (See Table below) as follows:

Surrogate Compound Recovery Action for Pesticides

Criteria	Action	
	Detected Target Compounds	**Non-Detected Target Compounds**
%R > 200%	J	No qualification
150% < %R ≤ 200%	J	No qualification
30% ≤ %R ≤ 150%	No qualification	
10% ≤ %R < 30%	J	UJ
%R < 10% (sample dilution not a factor)	J	R
%R < 10% (sample dilution is a factor)	Use professional judgment	
RT out of RT window	Use professional judgment	
RT within RT window	No qualification	

YES NO N/A

Note: Blank analysis having surrogates out of specification:

The reviewer must give special consideration to the validity of associated samples. Basic concern is whether the blank problems represent an isolated problem with the blank alone or whether there is a fundamental problem with the analytical process. For example, if one or more samples in the batch show acceptable surrogate recoveries, the reviewer may choose to consider the blank problem to be an isolated occurrence.

ACTION: Note in the Data Assessment under Contract Problems/ Non-Compliance if the Lab did not perform reanalysis and reviewer’s judgment regarding blank problem.

3.5 Are there any transcription/calculation errors between raw data and Form IIs? ___ [ ] ___

ACTION: If large errors exist, ask the TOPO to obtain an explanation/resubmittal from the lab, make any necessary corrections and note errors in the data assessment.

4.0 Matrix Spike/Matrix Spike Duplicate Recovery (Form III)

Note: Data for MS/MSD will not be present unless requested.

4.1 Are the MS/MSD Recovery Forms (Form III BNA) present? [ ] ___ ___

4.2 Was the MS/MSD analyzed at the required frequency (once per SDG, or every 20 samples, whichever is more frequent)? [ ] ___ ___

ACTION: If any MS/MSD data are missing, take action as specified in section 3.1 above.

ACTION: No action is taken on MS/MSD data alone. However, using professional judgement, the validator may use the MS and MSD results in conjunction with other QC criteria and determine the need for some qualification of the data. If Any MS/MSD % recovery or RPD is out of specification, qualify data to include the consideration of the existence of interference in the raw data. Consideration include, but not limited to the following “Action”:

Matrix Spike/Matrix Spike Duplicate Action for Pesticides

Criteria	Action	
	Detected Spike Compounds	**Non-detected Spike Compounds**
%R or RPD > Upper Acceptance Limit	J	No qualification
20% ≤ %R < Lower Acceptance Limit	J	UJ

YES NO N/A

%R < 20%	Use professional judgment
Lower Acceptance Limit ≤ %R; RPD ≤ Upper Acceptance Limit	No qualification required

Note: If it can be determined that the results of the MS/MSD affects only the sample spiked, limit qualification to only this sample. However, use professional judgment when it is determined through the MS/MSD results that the laboratory is having systematic problem in the analysis of one or more analytes that affect all associated samples.

5.0 Blanks (Form IV)

5.1 Is the Pesticide Method Blank Summary (Form IV PEST) present for aqueous and soil samples? [ ] ___ ___

5.2 Frequency of Analysis: For the analysis of PEST TCL compounds, has a method blank been analyzed for each SDG or every 20 samples, whichever is more frequent? [ ] ___ ___

ACTION: If any blank data are missing, take action as specified above in section 3.1. If blank data is not available, reject "R" all associated positive data. However, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.

5.3 A separate Form IV should be present if part of an extraction batch required sulfur removal. In such cases some samples will be listed on two blank summary forms - once under the method blank, and once under the sulfur clean-up blank (PCBLK). Was this additional blank raw data and Form IV submitted when required? [ ] ___ ___

ACTION: If Form IV sulfur clean-up blank is missing, take action as specified in section 3.1 above.

5.4 Has a Pesticide instrument blank been analyzed at the beginning of every 12 hr. period following the initial calibration sequence (minimum contract requirement)? [ ] ___ ___

ACTION: If any blank data are missing, take action specified in Section 3.1.

5.5 Was the correct identification scheme used for all Pesticide blanks? (See page B-39, section 3.3.7.3 of SOM01.1 for further information) [ ] ___ ___

ACTION: Contact the TOPO to obtain resubmittals or make the required corrections on the forms.

YES NO N/A

Document in the Data Assessment under Contract Problems/Non-Compliance all corrections made by the validator.

5.6 Chromatography: Review the blank raw data chromatogram, quant. Reports and data system printout. Is the chromatographic performance (baseline stability) acceptable for each instrument? [ ] ___ ___

ACTION: Use professional judgement to determine the effect on the data.

5.7 Are all detected hits for target compounds in method, and field blanks less than the CRQL? [ ] ___ ___

ACTION: IF no, an explanation and laboratory's corrective actions must be addressed in the case SDG narrative. Contact TOPO to request from Lab. revised narrative and make a note in the Contract Problems/Non-Compliance section of the Data Assessment.

6.0 Contamination

NOTE: "Water blanks", "drill blanks", and distilled water blanks" are validated like any other sample, and are not used to qualify data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method/reagent or cleanup blanks contain positive hits for target pesticide compounds with values greater than the CRQL for that analyte? ___ [ ] ___

Note: The concentration of each target compound in the instrument blank must be less than the CRQL for that analyte.

ACTION: Make note in data assessment under Contract Problems/Non-Compliance if any blank contains hit above the CRQLs.

6.2 Do any instrument blanks contain positive Pesticide results with values greater than CRQLs? ___ [ ] ___

ACTION: Take the action specified in section 6.1.

6.3 Do any field/rinse blanks have positive Pesticide results? ___ [ ] ___

NOTE: All field blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for system monitoring compound, instrument performance criteria, spectral or calibration QC problems.

ACTION: Follow the directions in the table below to qualify results due to contamination. Use the largest value from all the associated

YES NO N/A

blanks. If any blanks are grossly contaminated, all associated sample data should be qualified unusable (R).

Blank Action for Pesticide Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Field, Sulfur Cleanup, Instrument	Detects	Not detected	No qualification required
	< CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification required
	= CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification required
	> CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank contamination	Report concentration of sample with a U
		≥ CRQL and ≥ blank contamination	No qualification required
	Gross contamination	Detects	Qualify results as unusable R

NOTE: Analytes qualified "U" for blank contamination are treated as "hits" when qualifying for calibration criteria.
Note: When applied as described in the table above, the contaminant concentration in the blank are multiplied by the sample dilution factor.

6.4 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

ACTION: Note in data assessment if there’s no associated field/rinse/equipment blank.

Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 Gas Chromatography with Electron Capture Detector (GC/ECD) Instrument Performance Check (Form VI-5 thru 10, Form VII-1)

7.1 Are the following Forms, chromatograms and data system printouts present?

	YES	NO	N/A
a.) Form VI Pest-5/Pesticide Resolution Check Mix	[_]	___	___
b.) Form VI Pest-6/Performance Evaluation Mixture	[_]	___	___
c.) Form VI Pest-7/Individual Standard Mixture A	[_]	___	___
d.) Form VI Pest-8/Individual Standard Mixture B	[_]	___	___
e.) Form VI Pest-9/Individual Standard Mixture C	[_]	___	___
f.) Form VI Pest-10/Individual Standard Mixture C	[_]	___	___
g.) Form VII Pest-1/Calibration Verification	[_]	___	___
h.) Were the appropriate GC columns used as specified on page D-11/Pest, sections 6.26.1.3 to 6.26.1.3.2 in SOM01.1?	[_]	___	___

7.2 The identification of a single component pesticide by GC method is based primarily on RT data. Were the following requirements met:

	YES	NO	N/A
a.) The chromatogram that results for PEM and Individual Standards Mixture analyses must display the analytes at > 10% full scale but < 100% full scale	[_]	___	___
b.) The baseline of the chromatogram must return to below 50% of full scale before the elution of alpha-BHC, and return to below 25% of full scale after the elution time of alpha-BHC and before the elution time of decachlorobiphenyl	[_]	___	___

NOTE: If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram, and if standard, blank, etc chromatogram needs to be replotted electronically to meet these requirements, both the initial chromatogram and the replotted chromatogram(s) must be submitted in the data package.

ACTION: If all single component pesticides (SCP) are not clearly displayed on chromatograms for all Individual Standard Mixtures and PEM, notify the TOPO to obtain resubmittal of the necessary data.

	YES	NO	N/A
7.3 Are there any transcription/calculation errors between raw data and the Forms?	[_]	___	___

ACTION: If large errors exist, take action specified in section 3.1 above.

7.4 <u>Resolution Check Mixture (Form VI Pest-5)</u>

YES NO N/A

This mixture is analyzed at the beginning of every initial calibration sequence. Were the following met: [ ] ___ ___

a.) If two Individual Standard Mixture (A and B) are used, the resolution is ≥ 60% in both GC columns or

b.) One Individual Standard Mixture C is used, the resolution between two adjacent peaks is ≥ 80% on the primary column and ≥ 50% on the secondary column.

ACTION: If no, follow the action in Action Table below.

7.5 Performance Evaluation Mixture (Form VI Pest-6)

This mixture is analyzed at the beginning (following the Resolution Check Mixture) and at the end of the initial calibration sequence. Were the following met? [ ] ___ ___

a.) The resolution between any two adjacent peaks in the initial and continuing calibration verification must be ≥ 90% on each column.

b.) The % breakdown of 4,4'-DDT and Endrin in the PEMs must be ≤ 20.0% on each column and the combined % breakdown for 4,4'-DDT and Endrin in the PEMs must be ≤ 30.0% on each column.

ACTION: IF no, take action as specified in Action Table below.

7.6 Mid-Point Individual Standard Mixture (A and B) or (C)

The resolution capabilities of the GC/ECD system used will dictate which Individual Standard Mixture can be used. This is determined by analysis of the Resolution Check Mixture (RCM) to see if the RCM criteria were met (see section 7.4 above). Were the following criteria met? [ ] ___ ___

a.) Mid-Point Individual Standard Mixture A and B:
See section 7.4 a.) Above

b.) Mid-Point Individual Standard Mixture C:
See section 7.4 b.) Above

ACTION: If no, take action as specified in the following Table.

Table: Gas Chromatography with Electron Capture Detector (GC/ECD) Instrument Performance Check Action

Criteria [(Individual Standard Mixture (A and B)]	Criteria (Individual Standard Mixture C)	Action
Resolution Check Mixture % Resolution <60.0%	Resolution Check Mixture % Resolution <80.0% (primary column) % Resolution <50.0% (secondary column)	Detects: JN Non-detects: R
PEM % Resolution <90.0%		Detects: JN Non-detects : R
PEM: 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is detected		Detects for 4,4'-DDT: J Detects for 4,4'-DDD: J Detects for 4,4'-DDE: J
PEM: 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is not detected		Non-detects for 4,4'-DDT: R Detects for 4,4'-DDD: JN Detects for 4,4'-DDE: JN
PEM: Endrin % Breakdown >20.0% and Endrin is detected		Detects for Endrin: J Detects for Endrin aldehyde: J Detects for Endrin ketone: J
PEM: Endrin % Breakdown >20.0% and Endrin is not detected		Detects for Endrin: R Detects for Endrin aldehyde: JN Detects for Endrin ketone: JN
PEM: Combined % Breakdown > 30.0%		Apply qualifiers as described above considering degree of individual breakdown
Mid-point Individual Standard Mixtures (A and B) % Resolution <90.0%	Mid-point Individual Standard Mixture (C) % Resolution <80.0% (primary column) Mid-point Individual Standard Mixture (C) % Resolution <50.0% (secondary column)	Detects: JN Non-detects: R
PEM analysis not performed at the required frequency *		All results: R
Mid-point Individual Standard Mixtures analysis not performed at the required frequency **		All results: R

* The PEM is analyzed at the beginning (following the Resolution Check Mixture) and at the end of the initial calibration.

** Mid-point Individual Standard Mixture A and B: Analyzed as part of the initial calibration. The mid-point INDA and INDB must bracket one end of each 12-hour analytical period.

Mid-point Individual Standard Mixture C: Analyzed as part of the initial calibration. The mid-point INDC must bracket one end of each 12-hour analytical period.

YES NO N/A

7.7 Initial Calibration (Form VI Pest-2, Form VI Pest-3, Form VI Pest-3)

Were the Initial Calibration %RSD criteria met? [ ] ___ ___

ACTION: If no, qualify the data according to the following table:

Initial Calibration Action for Pesticide analyses

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
Initial calibration is not performed or not performed in proper sequence	Use Professional Judgment and notify Contract Lab Program (CLP) Project Officer	
%RSD exceeds allowable limits *	J	No qualification
%RSD within allowable limits *	No qualification	

* %RSD < 20.0% for single component target compound except alpha-BHC and delta-BHC.
%RSD < 25.0% for alpha-BHC and delta-BHC.
%RSD < 30.0% for Toxaphene.
%RSD < 30.0 for surrogates (tetrachloro-m-xylene and decachlorobiphenyl).

7.8 Continuing Calibration Verification (CCV) (Form VII)

Were the Absolute Retention Time (RT) for each Single Component Pesticide (SCP) and surrogate in the PEM and mid-point concentration of Individual Standard Mixtures (A and B) or (C) within the RT window determined from the initial calibration? [ ] ___ ___

ACTION: If no, use the following table to qualify pesticide analytes:

Continuing Calibration Verification (CCV) Action for Pesticides Analyses

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
RT out of RT Window	Use professional Judgment *	
Percent Difference not within limits **	J	UJ
Time elapsed is greater than acceptable limits ***	R	

YES NO N/A

Percent Difference, time elapsed and RT are within acceptable limits	No qualification

* For peaks close to the expected RT window of the pesticide of interest, the reviewer may take additional effort to determine if sample peaks represent the compound of interest. For example, the reviewer can examine the data package for the presence of three or more standards containing the pesticide of interest that were run within the analytical sequence during which the sample was analyzed. If three or more standards are present, the RT window can be re-evaluated using the mean RT of the standards. If the peak falls within the revised window, qualify detects as “JN”. Peaks that cannot be resolved with the revised window, qualify as unusable “R”.

** The Percent Difference (%D) for each of the SCP and surrogates in the PEM used for CCV must be greater than or equal to -25.0% and less than or equal to 25.0%. The %D between the Calibration Factor (CF) for each of the SCP and surrogates in the Calibration Verification Standard (CS3) and the mean calibration factor from the initial calibration must be greater than or equal to -20.0% and less than or equal to 20.0%. This criteria also applies to Toxaphene.

*** No more than 14 hours may elapse from the injection of the instrument blank that begins an analytical sequence (opening CCV) and the injection of either the PEM or mid-point concentration of the Individual Standard Mixtures (A and B) or (C) that ends an analytical sequence (closing CCV). No more than 12 hours may elapse from the injection of the instrument blank that begins an analytical sequence (opening CCV) and the injection of the last sample or blank that is part of the same analytical sequence. No more than 72 hours may elapse from the injection of the sample with a Toxaphene detection and the Toxaphene Calibration Verification Standard (CS3).

8.0 <u>Analytical Sequence Check (Form VIII-Pest)</u>

8.1 Is Form VIII-Pest present and complete for each column and each period of analyses? [ ] ___ ___

ACTION: If no, take action as specified in section 3.1

8.2 Was the proper analytical sequence followed for each initial calibration and subsequent analyses, and all standards analyzed at the required frequency for each GC/ECD instrument used? [ ] ___ ___

ACTION: If no, use professional judgment to determine the severity of the effect on the data and qualify accordingly. Generally, the effect is negligible unless the sequence was grossly altered and/or the calibration was out of QC limits.

8.3 Are the surrogate retention time (RT) from the initial calibration for TCX and DCB provided on Form VIII-Pest? [ ] ___ ___

YES NO N/A

ACTION: If no, take action as specified in section 3.1

8.4 Was the asterisk (*) applied to the RT of any blanks, samples, standards, MS/MSD, and LCS that did not meet the QC Limits of ± 0.05 minutes for TCX (tetrachloro-m-xylene) and ± 0.10 minutes for DCB (decachlorobiphenyl)? [ ] ___ ___

ACTION: If any data are missing, take action specified in 3.1 above.

If no, use professional judgment to determine the severity of the effect on the data and qualify accordingly. Document in the data assessment under Contract Problems/Non-Compliance.

9.0 Florisil Cartridge (Form IX Pest-1)and Gel Permeation Chromatography (GPC) (Form IX Pest-2) Performance Check

9.1 Is Form IX Pest-1 present and complete for each lot of cartridge used? [ ] ___ ___

Note: Florisil cartridge cleanup is mandatory for all extracts

Are all samples listed on the Pesticide Cartridge Form? [ ] ___ ___

ACTION: If no, take action specified in section 3.1

9.2 Are the percent recoveries of the target pesticides and surrogates in the Florisil performance check within 80-120% and the recovery of 2,4,5-Trichlorophenol is less than 5%? [ ] ___ ___

If the Florisil Cartridge Performance Check criteria were not met, qualify the data as follows:

Florisil Cartridge Performance Check Actions

Criteria	ACTION	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
%R > 120% (pesticide target compounds)	J	No qualification
80% ≤ %R ≤ 120%	No qualification	

10% ≤ %R < 80% (pesticide target compounds)	J	UJ
%R < 10% (pesticide target compounds)	J	R
%R > 5% (2,4,5-Trichlorophenol)	Use professional judgment *	

* Check sample chromatogram for interferences

9.3 If GPC cleanup was performed on aqueous samples (<u>mandatary</u> for all <u>soil</u> samples), is Form IX Pest-2 present? [ ] ___ ___

Are all soil samples listed on Form IX Pest-2? [ ] ___ ___

ACTION: If no, take action as specified in section 3.1.

9.4 Were the percent recoveries of the pesticides in the GPC continuing calibration verification solution within 80 to 110%? [ ] ___ ___

ACTION: If no, qualify the sample data as follows:

<u>Gel Permeation Chromatography (GPC) Performance Check Actions</u>

Criteria	**Action**	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
%R < 10% (pesticide target compounds)	J	R
10% ≤ %R < 80%	J	UJ
80% ≤ %R ≤ 110%	No qualification	
%R > 110% (pesticide target compounds)	J	No qualification

10.0 Laboratory Control Samples (LCS)

10.1 LCSs orovide information on the accurracy of the analytical method and laboratory performance.

LCS Spike Compound	**Recovery Limits (%)**	**LCS Spike Compound**	**Recovery Limits (%)**
gamma-BHC	50 - 120	Endosulfuran sulfate	50 - 120
Heptachlor epoxide	50 - 150	gamma-Chlordane	30 - 130
Dieldrin	30 - 130	Tetra-m-xylene (surrogate)	30 - 150

YES NO N/A

4,4'-DDE	50 - 150	Decachlorobiphenyl (surroagte)	30 - 150
Endrin	50 - 120		

10.2 Were the above recoveries met? [ ] ___ ___

Action: If no, qualify the sample data as follows:

Laboratory Control Sample (LCS) Actions

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Assoicated Compounds**
%R > Upper Acceptance Limit	J	No qualification
%R < Lower acceptance Limit	J	R
Lower Acceptance Limit ≤ %R ≤ Upper Acceptance Limit	No qualification	

11.0 Pesticide Identification (Form X Pest-1, Pest-2)

11.1 Is Form X (Pest-1 & Pest-2) complete for every sample in which pesticide was detected? [ ] ___ ___

ACTION: Take action as specified in section 3.1 above.

11.2 Are all sample chromatograms properly scaled, attenuated, etc. as required for proper identification of pesticides? (Refer to SOM01.1 sections 11.3.9 -11.3.9.7, pages D65-66) [ ] ___ ___

Note: Proper identification of pesticides depends on clear, legible presentation of the raw data. Pesticide peaks must be between 10-100% and Toxaphene between 25-100% of full scale. For any sample or blank, the baseline of the chromatogram must return below 50% of full scale before the elution time of alpha-BHC and return to 25% of full scale after the elution time of alpha-BHC and before the elution of decachlorobiphenyl.

ACTION: If retention times (RT) or peak apex cannot be verified, contact TOPO to obtain rescaled chromatograms from the lab.

11.3 Are there any transcription/calculation errors in Form I and Form X Pest-1, Form X Pest-2? [ ] ___ ___

ACTION: Take action as specified in section 3.1 above.

YES NO N/A

11.4 Are the RTs of pesticides within the established RT window for analyses on both columns? [ ] ___ ___

Was the GC/MS confirmation provided for pesticides concentration > 10 ug/ml in final extract? [ ] ___ ___

ACTION: Use professional judgement to qualify positive results which were not confirmed by GC/MS analysis. Check the semivolatile TIC data for presence of pesticides.

11.5 Is the per cent difference (%D) calculated for positive results on both columns < 25%? [ ] ___ ___

ACTION: The reviewer must check columns for peak interferences for the positive hits. Qualify the pesticide according to following Table:

<u>Action on Qualifying Positive Pesticide Results</u>

Percent Differences	**Qualifier**
0 - 25%	None
26 - 70%	“J”
71 - 100%	“JN”
101 - 200% (No Peak Interferences)	“R”
101 - 200% (Interferences detected)*	“JN”
> 50% (Pesticide value < CRQL)**	“U”
> 200%	“R”

* When interferences is detected on either column, qualify the data as “JN”

** When the pesticide value is below CRQL and %D > 50%, raise the value to CRQL and qualify “U”, undetected.

12.0 <u>Target Pesticide List (TCL)</u>

12.1 Are the Pesticide Analysis Data Sheets (Form I Pest) present with required header information on each page for samples, MS/MSD (if required), method and instrument blanks (per column & analysis)? [ ] ___ ___

	YES	NO	N/A
12.2 Is the chromatographic performance acceptable with respect to baseline stability, full-scale attenuation, peak shape/resolution?	[ ]	___	___

ACTION: If no, take action specified in section 3.1 above.

13.0 <u>Compound Quantitation and Reported Detection Limits</u>

	YES	NO	N/A
13.1 Are there any transcription/calculation errors in the Form I results? Check at least two positive results. Were any errors found?	[ ]	___	___

ACTION: If errors were found, take action as specified in section 3.1 above.

	YES	NO	N/A
13.2 Are the contract required quantitation limits (CRQL) adjusted to reflect sample dilution?	[ ]	___	___

ACTION: If errors exist, take action as specified in section 3.1 above.

ACTION: When a sample is required to be diluted, the lowest CRQL is used (unless a QC exceedance dictates the use of the higher CRQL from the diluted sample). Replace concentration which exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with the result from the diluted sample. Specify which Form I to use. Use a red pencil and draw a red "X" across the entire page of all Form I's that should not be used, including those in the data summary package.

At the top or bottom of the Forms, write with red pencil, "DO Not Use".

Note: If the sample dilution factor (DF) is greater than 10, an additional 10 times more <u>concentrated</u> than the diluted sample extract must be analyzed and reported with the sample data. If the DF is less or equal to 10, but greater than 1, the results of the original undiluted analysis must also be reported (see SOM01.1/section 10.4.3.5/page D-56).

ACTION: IF the above requirement was not met, contact the TOPO to obtain an explanation/resubmittal from the lab and make a note in the Data Assessment under Contract Problems/Non-Compliance section.

	YES	NO	N/A
13.3 For non-aqueous samples, were the percent moisture < 70%?	[ ]	___	___

Action: If the % moisture $\geq$ 70.0% and < 90.0%, qualify detects as "J" and non-detects as approximated "UJ" If the % Moisture $\geq$ 90%, qualify detects as "J" and non-detects as "R"

14.0 <u>Field Duplicates</u>

14.1 Were any field duplicates submitted for Pesticide analysis? [ ] ___ ___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between duplicate results must be addressed in the reviewer narrative. If large differences exist, contact the TOPO to confirm identification of field duplicates with the sampler.

Definitions

CCS - contract compliance screening
CF - Calibration Factor
CLASS - Contract Laboratory Analytical Services Support
CLP - Contract Laboratory Program
CRQL - Contract Required Quantitation Limit
GC/ECD - Gas Chromatography/Electron Capture Detector
kg - kilogram
µg - microgram
ℓ - liter
mℓ - milliliter
PEM - Performance Evaluation Mixture
QC - quality control
RAS - Routine Analytical Services
RPD - Relative Percent Difference
RRF - Relative Response Factor
$\overline{RRF}$ - Average Relative Response Factor (from initial calibration)
RRT - Relative Retention Time
RSD - Relative Standard Deviation
RT - Retention Time
RSCC - Regional Sample Control Center
SCP - Single Component Pesticide
SDG - Sample Delivery Group
SOP - standard operating procedure
SOW - Statement of Work
PEST - Pesticides
TCL - Target Compound List
TCLP - Toxicity Characteristics Leachate Procedure
TIC - Tentatively Identified Compound
TPO - Technical Project Officer
VTSR - Validated Time of Sample Receipt
TOPO - Task Order Project Officer

References

1. USEPA Contract Laboratory Program of Work for Organic Analysis Multi-Media, Multi-Concentration, SOW/CLP/SOM01.1, October 2004
2. National Functional Guidelines for Superfund Organic Methods Data Review January 2005